State of New Jersey

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P.O. Box 7176
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AUG 2 1 1997

Re: Draft Work Plan for Supplemental Ecological Report

Dear Mr. Smith:

The New Jersey Department of Environmental Protection (NJDEP) is in receipt of the Draft Work Plan for the Supplemental Ecological Report (SER) dated June 19, 1997. The referenced document was reviewed in accordance with Risk Assessment Guidelines for Superfund, Volume II, Environmental Evaluation Manual (EPA/540/1-89/001), NJDEP policy, and other State and Federal guidance. Upon review of the subject document, NJDEP has a few comments. The comments have been attached for your convenience.

If you have any questions regarding this letter, please do not hesitate to contact me at (609) 633-1494.

Sincerely,

Donna L. Gaffigan

Donna L. Gaffigan, Case Manager
Bureau of Federal Case Management

Enclosures

C: William Hanrahan, BGWPA
Steven Byrnes, BEERA
Dr. Edward Demarest, BEERA
William Lawler, USEPA
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DRAFT WORK PLAN
SUPPLEMENTAL ECOLOGICAL REPORT (SER)

June 19, 1997

1. Section 1.1 - NAWC Background, p. 1-1

   Figure 1-1 is referenced in the second paragraph, however, no figure is so designated. A Figure 17-1 is provided which may be the Figure intended since Figure 17-1 is not referenced in the text. This must be clarified.

2. Section 1.4 - Approach Overview, p. 1.4

   In addition to the May 19, 1994 NJDEP Technical Requirements for Site Remediation (Tech Regs), N.J.A.C. 7:26-E, et seq., activities conducted pursuant to the SER must be in full compliance with the Tech Reg Ammendments effective July 18, 1997 (see N.J.R. 2278(b)).

3. Section 3.2.1 - Sampling Locations - Reference Areas, p. 3-1

   Figure 3-1 is referenced in the second paragraph, however, no figure is so designated. A Figure 17-2 is provided which may be the Figure intended since Figure 17-2 is not referenced in the text. This must be clarified.

4. Section 3.3.2 - Surface Sediment, p. 3-3

   Grab samples are proposed for surface sediment. NJDEP specifies that surficial freshwater sediments be obtained from the 0-6" interval (primary biotic zone). Samples should be collected within this interval with the specific depth (0-3", 0-4", etc.) reported if different from 0-6".

5. Section 3.4.1 - Analytical Methods - Surface Water, p. 3-4

   a. In Table 3-2 it is proposed that surface water be analyzed for hardness. Hardness must be analyzed as mg/L CaCO3 as per the New Jersey Surface Water Quality Standards (NJSWQS) and Federal Surface Water Quality Criteria (FSWQC).

   b. It is proposed that surface water samples are filtered for metals analysis. NJDEP requires that *unfiltered* surface water samples be analyzed for metals. Filtered samples are acceptable in addition to the unfiltered samples.

   c. The surface water samples must also be analyzed for total selenium for comparison to both the chronic and acute aquatic standards (NJSWQS default to FAWQC).

6. Section 3.4.2 - Analytical Methods- Surface Semiment, p. 3-4

   For sediments, NJDEP modified Method 3060, USEPA SW846 2nd Edition and Method 7196A, USEPA SW846 3rd Edition must be used for alkaline digestion and hexavalent chromium analysis, respectively. A copy of each method has been attached for your convenience.

7. Section 4.1.2 - Screening Values - Surface Water, p. 4-2

   a. In Table 4-1, it is proposed to use "NJ Surface Water *Criteria* Standards" as screening values. It must be correctly stated in the table that the NJ values are *standards*, not *criteria* as are the Federal values.

b. In Table 4-1, it is proposed that the "EA Reporting Limit" for aluminum be 200 ug/l. This reporting limit is too high and must be equal to or lower than the screening value of 87 ug/l.

c. In Table 4-1, the stated Minimum Detection Limits (MDLs) for inorganic mercury and silver are 0.2 ppb and 4.0 ppb, respectively. The NJSWQS for chronic aquatic effects from mercury is 0.012 ppb and the acute (there is no chronic listed) effects level for silver is 3.4 ppb. NJDEP will accept the proposed MDLs provided that the Instrument Detection Limits (IDL) are at or below these standards.

8. Section 4.1.2 - Screening Values - Surface Water, p. 4-2

In Table 4-2, it is proposed that the "EA Reporting Limit" for arsenic be 10.0 mg/kg. This reporting limit is too high and must be equal to or lower than the screening value of 6.0 mg/kg.

9. Section 4.3 - COPC Bioavailability, p. 4-4

a. In the first paragraph it is stated that the identified Constituents of Potential Concern (COPC) were not necessarily measured in the form that the screening level is representing as toxic. Bullets 1 and 3 indicate that hexavalent chromium and methyl mercury levels will be analyzed in sediments. However, it should not be assumed, for example, that a chromium screening value is necessarily based on the hexavalent form. The basis and background document for a screening scheme should be consulted to determine the chemical form upon which a value is based.

b. The last paragraph states that if access to Gold Run Pond 1 is not granted, then it is proposed to assess availability/toxicity by comparing reference area analytical results to relationships found in the literature to interpret the finding for Gold Run. Please be advised that the data for Gold Run Pond 1 must be collected from the pond itself.

10. Section 4.4 - Food Chain Model for Selenium, p. 4-5

In the daily dose calculations it is proposed to use input parameters for water/food ingestion rates and body weights from the 1994 Remedial Investigation Report. NAWC must use input parameters for the 3 species as provided in the EPA Wildlife Exposure Factors Handbook, referenced below. This is the reference used both by EPA Region II and NJDEP. This is also the recommended guidance in the New Jersey Technical Requirements for Site Remediation (see N.J.A.C.7:26E-4.7).

REFERENCES

US Environmental Protection Agency. 1993. Wildlife Exposure Factors Handbook. Volume I. EPA 600/R-93/187a.

NJDEP MODIFIED METHOD 3060
USEPA SW846 2nd Edition
ALKALINE DIGESTION

1.0 Scope and Application

1.1 This method is used to prepare samples for the determination of the concentration of hexavalent chromium in solid samples such as soil, sludge, sediment, debris, brick, concrete, and precipitate.

2.0 Summary of Method

2.1 This method uses basic digestion of the solid sample to solubilize both water-insoluble and water-soluble hexavalent chromium compounds.

2.2 The sample is extracted with hot, 3% sodium carbonate - 2% sodium hydroxide solution to dissolve Cr(VI).

3.0 Interferences

3.1 Solid samples containing high buffering capacity may require additional digestion solution (see section 5.5) to properly digest the sample.

4.0 Apparatus

4.1 Class A glassware: 250-ml beaker (with watch glass covers), 100-ml graduated cylinder with stopper, 1,000 ml and 100 ml volumetric flasks, and 250 ml flasks.

4.2 Vacuum filtration apparatus.

4.3 Filter paper (0.45um and 0.1um), record in laboratory notebook the filter brand used.

4.4 Hot plate or water bath with auto stirring capability.

4.5 Volumetric Class A Pipettes: assorted sizes, as necessary.

4.6 pH meter.

4.7 Calibrated balance.

4.8 Thermometer (NIST Certified).

5.0 Reagents

5.1 Nitric acid: HNO_3, concentrated, analytical reagent grade.

5.2 Sodium carbonate: Na_2CO_3, anhydrous, analytical reagent grade.

5.3 Sodium hydroxide: NaOH, analytical reagent grade.

5.4 Potassium dichromate: $K_2Cr_2O_7$, analytical reagent grade.

5.5 Digestion solution: Dissolve 20.0 g sodium hydroxide and 30.0 g sodium carbonate in Type I water in a 1-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle and prepare fresh monthly. Also, the pH of the digestion solution must be checked before using and have a pH of 11.5 or greater.

5.6 Potassium dichromate spiking solution (1 ml = 1 mg Cr): Dissolve 2.829 g of dried potassium dichromate in Type I water in a 1-liter volumetric flask and dilute to the mark; alternatively a 1,000 mg/l chromium stock solution can be used (Fisher AAS Standard or equivalent).

6.0 Sample Handling and Preservation

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in N.J.A.C. 7.26 E-3.6 (Site Investigations).

6.2 To retard the chemical activity of hexavalent chromium, the solid sample must be stored at 4° C until analyzed.

6.3 Since the stability of hexavalent chromium is not fully understood at this time, all samples must be digested and analyzed within 48 hours of sample collection. The maximum holding time prior to analysis of the samples is 48 hours unless otherwise directed by NJDEP. The 48 hour holding time begins at the time of sample collection in the field.

6.4 All samples in a batch of no more than 20 samples shall be analyzed within one hour after the last sample has been digested, filtered, and pH adjusted.

7.0 Procedure

7.1 Place 2.5 g ± 0.25 g of the sample into a clean and labeled 250-ml beaker.

7.2 Add 50 ml of digestion solution (see section 5.5). Cover each beaker with a watch glass. Use a stirring bar for continuous stirring for five (5) minutes at a minimum. Then, heat the sample and maintain a temperature range of 90-95 °C with constant stirring for 30 to 60 minutes. Do not allow sample to go to dryness, as hexavalent chromium may be lost due to side reactions in the sample matrix.

7.3 Gradually cool the solution to room temperature and transfer it quantitatively to the filtration apparatus with Type I water rinses and filter through a 0.45um membrane filter. Rinse the inside of the filter flask and filter pad with Type I water and transfer the filtrate and the rinses to a clean and labeled 250 ml beaker.

7.4 The analyst shall start the color development and measurement procedure (NJDEP Modified Method 7196A USEPA SW846 3rd Edition), within one hour after the last sample in the batch has been filtered and pH adjusted. Just prior to analysis, place a magnetic stirring bar into the beaker, place the beaker on a stirrer and, with constant stirring, slowly add concentrated nitric acid to the beaker in small aliquots. Adjust the pH of the solution to 7.5 ± 0.5. The pH should be monitored with a pH meter. If the pH drops below 7.0 discard the solution and redigest. Caution: carbon dioxide and HNO_3 fumes will be evolved. This step should be performed in a fume hood.

7.5 Remove the stirring bar and rinse it, collecting the rinsate in the beaker. Transfer quantitatively the contents of the beaker to a 100 ml graduated cylinder with stopper or a 100 volumetric flask and adjust the sample volume to the mark with Type I water.

7.6 If the aliquot is brown, opaque, or a yellow color with intensity greater than the highest standard, the analyst must use the least dilution necessary to bring the concentration within the calibration range and record the dilution in the laboratory notebook.

7.7 Record the color and characteristics of the aliquot in the laboratory notebook both before and after dilution, if necessary.

7.8 Determine the concentration of hexavalent chromium by using NJDEP Modified Method 7196A USEPA SW846 3rd Edition.

8.0 Quality Control

8.1 All quality control (QC) data shall be maintained and available for easy reference or inspection. The following QC analyses must be performed per batch of no more than twenty (20) field samples of similar matrix and concentration.

8.2 One pre-digestion spike sample (spike at a concentration of 0.5 mg/L).

8.3 One duplicate laboratory sample and perform the duplicate analysis on the same sample that a prespike was performed.

8.4 One preparation/reagent blank.

8.5 One post verification spike sample (spike at a concentration of twice that of the original sample result or 150 ug/L, whichever is greater). Note: Do not spike this sample until after the analysis of the original sample and perform the post verification spike analysis on the same sample that a prespike was performed.

8.6 To check for possible losses of hexavalent chromium during digestion, digest and filter a mid-point hexavalent chromium calibration check standard (0.5 mg/L) and calibration blank by the same procedure as the sample(s).

NJDEP MODIFIED METHOD 7196A
USEPA SW846 3rd Edition
CHROMIUM, HEXAVALENT (COLORIMETRIC)

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of hexavalent chromium Cr(VI) in aqueous and digested solid samples.

1.2 The method may be used to analyze samples containing from 0.10 to 2 mg of Cr(VI) per L.

1.3 Solid samples must be digested first according to NJDEP Modified Method 3060 USEPA SW846 2nd Edition. The digestate shall be analyzed by the method contained herein.

2.0 SUMMARY OF METHOD

2.1 Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color of unknown composition is produced. The reaction is very sensitive, as the absorbancy index per gram atom of chromium is about 40,000 at 540 nm. Addition of an excess of diphenylcarbazide yields the red-violet product, and its absorbance is measured photometrically at 540 nm.

3.0 INTERFERENCES

3.1 The hexavalent chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the hexavalent chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can generally be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of hexavalent chromium will not generally cause interference.

3.2 Iron in concentrations greater than 1 mg/L may produce a yellow color, but the ferric iron color is not strong and difficulty is not normally encountered if the absorbance is measured photometrically at the appropriate wavelength (i.e., 540 nm)

3.3 Reducing substances such as organic matter or sulfides can convert hexavalent chromium to trivalent chromium. These reactions may occur in the natural environment and during the digestion and measurement procedures.

4.0 APPARATUS AND MATERIALS

4.1 Colorimetric equipment: One of the following is required: Either a spectrophotometer, for use at 540 nm, providing a light path of 1 cm or longer, or a filter photometer, providing a light path of 1 cm or longer and equipped with a greenish-yellow filter having maximum transmittance near 540 nm.

4.2 Class A glassware and pipets: 50, 100 and 250 ml beakers, 50 ml graduated cylinder, 100 ml volumetric flask and assorted pipets.

4.3 Filter paper (0.45 um and 0.1 um), record in laboratory notebook the filter brand used.

4.4 Vacuum filtration apparatus.

4.5 pH meter.

5.0 REAGENTS

5.1 Potassium dichromate stock solution: Dissolve 2.829g of dried potassium dichromate, $K_2Cr_2O_7$ (analytical reagent grade), in Type I water and dilute to 1 liter (1 ml = 1 mg Cr); alternatively a 1,000 mg/L chromium stock solution can be used (Fisher AAS standard or equivalent).

5.2 Sulfuric acid, 10% (v/v): Dilute 10 ml of distilled analytical reagent grade or spectrograde quality sulfuric acid, H_2SO_4, to 100 ml with Type I water.

5.3 Diphenylcarbazide solution: Dissolve 250 mg 1,5-diphenylcarbazide in 50 ml analytical reagent grade acetone. Store in a brown bottle. Discard when the solution becomes discolored or monthly whichever comes first.

5.4 Acetone (analytical reagent grade): Avoid or redistill Acetone that comes in containers with metal or metal-lined caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in N.J.A.C. 7:26E - 3.6 (Site Investigations).

6.2 Since the stability of Cr(VI) is not fully understood at this time, all samples must be digested and analyzed within 48 hours of sample collection. The maximum holding time prior to analysis of the samples is 48 hours unless otherwise directed by NJDEP. The 48 hour holding time begins at the time of sample collection in the field.

6.3 For all solid samples, the analyst shall start the color development and measurement procedure within one hour after the last sample in the batch has been filtered and pH adjusted.

6.4 All samples shall be stored at $4^{o}C$ upon field collection. No acid may be added to aqueous samples.

7.0 PROCEDURE

7.1 COLOR DEVELOPMENT AND MEASUREMENT:

7.1.1 For all samples (field samples and QC samples) transfer quantitatively 45.0 ml of the sample to be analyzed to a 100 ml beaker.

7.1.2 Add 1.0 ml of diphenylcarbazide solution and mix well.

7.1.3 Add 10% sulfuric acid to each sample and mix well after each addition to adjust the pH to a range of 1.6-2.2. Test the pH of each sample with a pH meter when the effervescence has ceased and record the final reading in the laboratory notebook.

7.1.4 If the samples are turbid, filter them using a 0.45 um membrane filter. If samples are still turbid, the analyst(s) may filter the samples again using a 0.1 um membrane filter.

7.1.5 Dilute the sample quantitatively to a final volume of 50 ml with Type I water, mix well, and let stand 5 to 10 minutes for full color development. Then, take a reading of the sample on the instrument being used.

7.1.6 Take 10 mls of the original sample without the diphenylcarbizide addition to use for background correction. Add sulfuric acid and mix well after each addition until the pH is adjusted to a range of 1.6 to 2.2. Test the pH of each sample with a pH meter when the effervescence has ceased and record the final reading in the laboratory notebook. Do not add diphenylcarbazide.

7.2 PREPARATION OF CALIBRATION CURVE FOR SOLID SAMPLES:

7.2.1 Calibration standards must be prepared fresh daily or each time the analysis is run. For instrument calibration, prepare a calibration blank and at least four calibration standards ranging from 0.10 to 2.0 mg/L Cr(VI). To check for possible losses of chromium during digestion of a solid sample, prepare and analyze a mid-point calibration check standard following the same procedure as the solid sample by digesting and filtering the standard according to NJDEP Modified Method 3060 USEPA SW846 2nd Edition.

7.2.2 Calibration standards are prepared by adding 50 mls of digestion solution to 100 mls volumetric, spike accordingly with the stock Chromium solution, then fill to volume with Type I water.

7.2.3 Develop the color of the standards the same as for the solid sample(s). Refer to 7.1 for color development and measurement. Calculate the correlation coefficient, slope, and intercept for the calibration curve. If the calibration curve gives a correlation coefficient of less than 0.995, then prepare a new calibration curve.

7.3 PREPARATION OF CALIBRATION CURVE FOR AQUEOUS SAMPLES:

7.3.1 Calibration standards must be prepared fresh daily or each time the analysis is run. For instrument calibration, prepare a calibration blank and at least four calibration standards ranging from 0.10 to 2.0 mg/L Cr(VI) and a mid-point calibration check standard.

7.3.2 Calibration standards are prepared by diluting the stock Chromium solution with Type I water.

7.3.3 Develop the color of the standards the same as for the aqueous sample(s). Refer to 7.1 for color development and measurement. Calculate the correlation coefficient, slope, and intercept for the calibration curve. If the calibration curve gives a correlation coefficient of less than 0.995, then prepare a new calibration curve.

8.5 For aqueous and solid samples, run one duplicate sample per batch of no more than 20 samples of similar matrix type and concentration. A duplicate sample is a sample brought through the whole sample preparation and analytical process. The duplicate sample analysis should fall within an acceptance criteria of 20% RPD if the original and duplicate sample values are greater than or equal to 8 ppm. A control limit of $\pm$ 2 ppm must be used if either the original or duplicate value is less than 8 ppm.

8.6 For solid samples, run one predigestion spike sample per batch of no more than 20 solid samples of similar matrix type and concentration pursuant to NJDEP Modified Method 3060 USEPA SW846 2nd Edition Section 8.2. The predigestion spike analysis should fall within an acceptance criteria of 75-125%.

8.7 For aqueous and solid samples, run one post verification spike sample per batch of no more than 20 samples of similar matrix type and concentration. The post verification spike recovery must fall within an acceptance criteria of 85-115%.

8.8 A new 5 point (4 standards and a calibration blank) calibration curve must be performed each day. The calibration curve must have a correlation coefficient equal to or greater than 0.995.

9.0 CALCULATIONS

9.1 Percent Solids: Immediately following the weighing of the samples to be analyzed, add 5-10 grams of sample to a tared weighing dish. Weigh and record to the nearest 0.01g. Place the weighing dish plus the sample in a drying oven maintained at 105 degrees Centigrade. Dry the sample overnight (12-24 hours). Remove the sample from the oven and cool to room temperature in a dessicator. Weigh and record weight to the nearest 0.01g. Calculate percent solids by the formula below:

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

9.2 Calculate the hexavalent chromium result for each solid sample as follows:

$$\text{Hexavalent Chromium in mg/Kg} = \frac{A \times B \times E}{C \times D}$$

A = Concentration from the calibration curve in mg/L
B = Final digested volume in Liters
C = Wet sample weight
D = Percent solids
E = Dilution (if necessary)

9.3 Calculate the hexavalent chromium result for each aqueous sample as follows:

Hexavalent Chromium in mg/L = A x E

A = Concentration from the calibration curve in mg/L
E = Dilution (if necessary)

9.4 Sample spike percent recovery: The following calculation should be performed to determine percent recovery.

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

9.5 Calibration Check Recovery: The following calculation should be used to determine percent recovery of the calibration check standard (CCS).

$$\text{CCS Recovery} = \frac{\text{Digested CCS result}}{\text{True Value}} \times 100$$

9.6 Relative Percent Difference (RPD): The following calculation should be performed to determine RPD.

$$RPD = \frac{2 (S - D)}{S + D} \times 100$$

S = Original Sample Value
D = Duplicate Sample Value

7.5.3 If the result of the post verification spike indicates an interference, the sample shall be diluted and reanalyzed.

7.5.4 If post verification spike recovery of acidic samples yield recoveries of less than 85%, then reanalyze the sample to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be performed by first making an aliquot of the original water sample or the duplicate soil sample alkaline (pH 8.0-8.5) using 1 N sodium hydroxide and then respiking and reanalyzing according to step 7.1 (color development and measurement). If a post verification spike recovery of 85-115% is obtained in the alkaline aliquot of an acidic extract that initially was found to contain less than 1 mg/L Cr(VI), one can conclude that the analytical result has been verified. If the post spike recovery is still outside the criteria, the analyst should refer to step 7.5.5.

7.5.5 If the interference persists after sample dilution, an alternative method (such as USEPA SW846 3rd Edition Method 7197, Chelation/ Extraction) may be used but is not required.

8.0 QUALITY CONTROL

8.1 All quality control data shall be maintained and available for easy reference or inspection. Refer to Chapter One in the USEPA SW846 3rd Edition for more information.

8.2 Dilute samples (using the least dilution necessary) if they are more concentrated than the highest standard.

8.3 For aqueous and solid samples, run one preparation/reagent blank per batch of no more than 20 samples of similar matrix and concentration to determine if there is any laboratory contamination. The preparation/reagent blank must contain all the reagents and in the same volumes as used in the preparation of the samples. Also, the values for the preparation blank must not be subtracted from the associated samples.

8.4 Verify instrument performance with a separately prepared mid-point calibration check standard every 10 samples followed by a calibration blank. The mid-point calibration check standard and the calibration blank must also be analyzed at the beginning of the run and after the last sample. The mid-point calibration check standard must fall within the control limits of 90-110% of the true value.

7.4 ANALYTICAL SEQUENCE

7.4.1 Calibrate the spectrophotometer daily using a 1-cm cell at 540 nm.

7.4.2 Analyze a calibration blank followed by at least four calibration standards in graduated (0.10 - 2.0 mg/L) amounts.

7.4.3 Immediately after the instrument is calibrated, analyze the mid-point calibration check standard and calibration blank and then analyze all samples in the sequence described in 7.4.4 below. Analyze the sample followed by the corresponding background correction sample. Correct the absorbance reading of the sample by subtracting the absorbance of the background correction sample. Record in the laboratory notebook all absorbances (background, uncorrected, and corrected).

7.4.4 Verify instrument performance by running a mid-point calibration check standard every ten samples which includes the background corrected sample (20 readings) followed by the calibration blank. The calibration check standard and calibration blank must also be analyzed at the beginning of the run and after the last sample.

7.5 VERIFICATION:

7.5.1 For every sample batch analyzed, a post verification spike is required to ensure that neither a reducing condition nor chemical interference is affecting color development. After the analysis of the original sample aliquot, spike another 45.0 ml aliquot of the sample at 2 times the concentration found in the original sample aliquot or 150 ug Cr(VI)/liter whichever is greater. Then follow 7.1 for color development and measurement. The post verification spike addition is prior to the addition of the reagents in 7.1. To verify the absence of an interference, the post verification spike recovery must be between 85% and 115%.

7.5.2 If addition of the post verification spike will extend the concentration beyond the calibration curve, the post verification spike sample aliquot shall be diluted with Type I water to within the calibration range prior to post verification spike addition and addition of reagents. Calculated results shall be adjusted accordingly.